

Extending Phase Optimized Liquid Chromatography (POPLC) to Green Chromatography

Kai Chen¹; Maarten De Beer¹; Laure Hitzel²; Frédéric Lynen¹; Pat Sandra¹

¹Pfizer Analytical Research Center, Ghent, Belgium; ²Pfizer Global R&D, Sandwich, UK

Phase OPTimized Liquid Chromatography (POPLC) is a promising technique to enhance the selectivity for a given separation by a combination of different stationary phases. Previous work based on mobile phase systems of water / acetonitrile shows that POPLC has good performance in method development for the separation of complex mixtures. To meet the requirements of green chromatography and taking into consideration the present shortage and high price of acetonitrile, this work aims to extend the POPLC technique to the green mobile phase systems such as water / ethanol. The results show that gradient elution profiles of water / ethanol with POPLC can also obtain satisfactory selectivities and resolutions for all the components within acceptable analyzing time.

A test sample containing 7 sulphonamides, 3 xanthine alkaloids and 4 steroids was analyzed using a POPLC[®] Kit (Bischoff Chromatography). The flow rate was 0.5 mL/min and the column temperature was set to 50 °C. The basic isocratic tests consisted of three stages of proportion of ethanol in the mobile phase, i.e. 15%, 30%, 45%. All five stationary phases in length of 10 cm were employed to measure the *k* value for each component. According to the “prisma model”, the theoretical *k* value for each component on each possible combination of basic stationary segments could be predicted and the corresponding selectivity / resolution values were obtained.

Due to the different hydrophobicities of the sample components, xanthine alkaloids and some sulphonamides had a relative short retention time, which resulted in poor resolution even for the optimized combination of stationary phase segments. In addition, the steroids showed strong hydrophobicities and the total analysis time exceeded 1 h.

A linear gradient profile was therefore developed. Retention time of each component under gradient conditions was re-calculated based on all the possibilities of basic stationary segment combinations. The segment combination with the highest selectivity was selected and the 14 components showed baseline separation in less than 30 min.